Docket No.: IFF-21
R&D Code No.:

ADDITIONAL EXAMPLE CONTAINED HEREIN

IFF INVENTION DISCLOSURE COVER SHEET (Research & Development)

Preparer: Michael Popplewell

Date: [redacted]

Departmental Manager Comments:

While there are other matrix systems that could give similar results, many of these are covered by patent, could create significant processing difficulties, or are expensive to practice. Based on the early commercial interest and the numerous other possible uses, I recommend that we at least formally assure ourselves that we can use the system described, and possibly file a provisional application to give us time to fully assess utility.

Director of R&D

Patent Counsel; Comments concerning patentability:

Additional Comments (if necessary) by preparer after review by Patent Counsel:

DESCRIPTION OF INVENTION

Title of Invention: Stable Encapsulation System

This invention relates to a matrix system for encapsulation of flavor, fragrance, color, or other active ingredients which has better stability than traditionally used matrices in the moist and elevated temperature conditions encountered in many applications. The invention thus allows the flavor, fragrance, appearance, and function of products to be improved by incorporation of additives in a way that protects against dispersion or loss during the manufacturing or preparation process.

This IDF should be completed and attached to the INVENTION DISCLOSURE COVER SHEET for forwarding to the appropriate departmental manager for his approval. He, in turn, shall forward it to the Director of R&D who shall forward same to Patent Counsel with appropriate commentary.

1. Title: Stable Encapsulation System

   1.1. Preparer: Michael Popplewell

   1.2. Date of Preparation: [redacted]

   1.3. Suggested Inventorship: Wen Lou and Michael Popplewell

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2. Earliest date of commencement of actual work on invention (attach photostats of notebook pages): [redacted]

   2.1. Date on which concept of invention was first formulated (attach pertinent memoranda): [redacted]

   Note: The work begun in 1999 was targeted to a different end-use, and contained additional ingredients and processing steps to those outlined below. Work begun in 2000 constitutes the actual invention.

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3. Structures of compounds to be covered; and/or schematic diagram of reactions covered by process for producing these compounds.

   A formula containing carriers such as sugars, maltodextrin, fats and a required amount of hydroxypropyl cellulose with or without flavor/fragrance/active is subjected to high temperature (100 to 190 C) at low moisture conditions for a short period of time. Subsequently, the heat treated mass is cooled and sized to create solid flavoring particles or granules. These granules/particles will not dissolve or deform in hard candy applications that reach approximately 290° F at 90 - 98% total solids. Additionally, in other confectionery systems processed at 50 - 70% solids and 140 - 220° F (gummy candies, chewy candies) the products do not dissolve, or do so at a very slow rate, thus allowing them to survive the manufacturing process largely intact. It is hypothesized that the slower solubility will also improve flavor/active delivery in higher-moisture food systems, as well as delivery of fragrances/actives in non-food systems which undergo similar processing/preparation.

   Further, it is hypothesized that particles made by other means (e.g. spray drying, drum drying, coating) will show similar properties, and so should be included in the invention.

The specific formula ranges projected are:

- Sugars, maltodextrins, fats and other carrier materials 60 - 99.5%
- Flavor/Fragrance/Active 0 - 20%
- Hydroxypropyl cellulose 0.5 - 20%

The effective use of such low levels of HPC to control temperature/moisture stability is unexpected.

4. Uses of compounds or products prepared in #3 (attach evaluation sheets):

As an example of matrix functionality, the following experiment was performed:

- (3) versions of a cherry flavor were made via melt extrusion. Each contained from 3 – 5% flavor, 2% lake color blend (blend of Red #40 and Blue #1 lakes), and 5% acid blend (blend of citric and malic acid) as actives. Aside from the levels of hydroxypropyl cellulose described below, the balance of the formula consisted of a similar blend of maltodextrin (GPC 15 DE, M150),sucrose, cottonseed oil, and silicone dioxide.
  a. E128201 Contained no hydroxypropyl cellulose (Hercules Klucel GF)
  b. E128202 Contained 2% hydroxypropyl cellulose (Hercules Klucel GF)
  c. E 128203 Contained 3% hydroxypropyl cellulose (Hercules Klucel GF)

  Each formula was extruded under similar conditions, cooled, and then sized to yield –10/+14 granules.
- Each sample was evaluated for stability when processed in the following hard candy formulation and procedure:

IPC#	DESCRIPTION	WEIGHT (%)
198863	Sugar, Fine Granule	56.0
36480	Corn Syrup, 43 BE	27.0
230195	Tap Water	17.0
	Total	100.0

The sugar, corn syrup and water are weighed into a 400 mL beaker and stirred together. The ingredients are heated in a 1000 watt microwave for approximately 1 minute and 45 seconds, until the temperature reaches approximately 300^0 F. The beaker is removed from the microwave and briefly stirred. The extruded flavor is added and blended until uniform. The melt is then deposited into moulds and allowed to cool. Once cool, the product is removed from the moulds and evaluated.

- The results showed that E128201 was significantly melted in the candy, bleeding color and showing little in the way of particle retention. E128202 and E128203 re-

tained their shape and color in the candy, resulting in a clear candy with discrete particles. When consumed, the candies containing E128202 and E128203 gave a slightly rough surface as the product dissolved, indicating that the extruded flavors had a slightly slower dissolution rate than the outer candy.

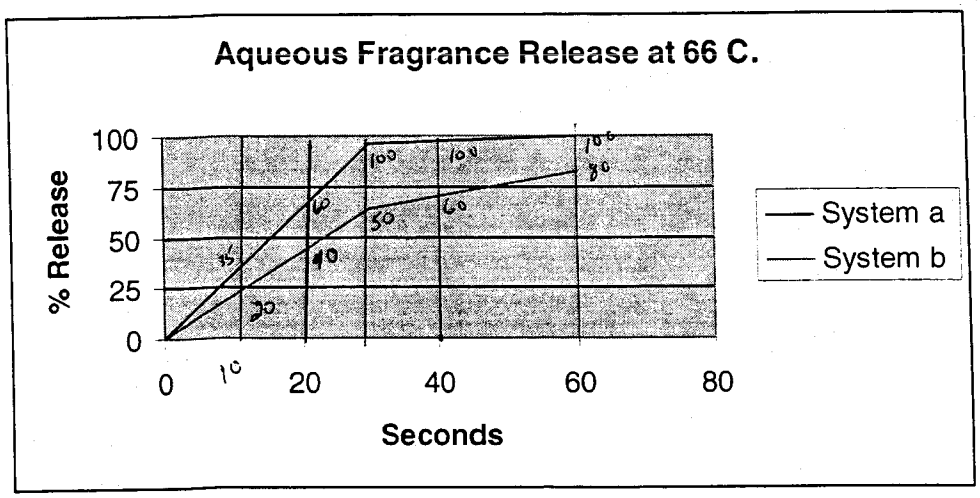
As another example of matrix functionality, the following experiment was performed:

(2) versions of fragrance extrudate were made. The compositions were as follows:

a. 10% of a fragrance consisting of equal parts of hexyl cinnamic aldehyde, lillial, and helional was encapsulated in a matrix consisting of maltodextrin, sucrose, maltose, and silicon dioxide. Lecithin was included as an emulsifier.

b. 12% of helional was encapsulated in a matrix consisting of 2% (total product weight) Klucel GF (Hercules), sucrose, maltodextrin, cottonseed oil, distilled monoglyceride, and silicon dioxide. Lecithin was used as an emulsifier.

Particles screened between 30 and 40 US mesh were dissolved in 66 C. water at a level of 0.5%. The dissolution was monitored via spectrophotometric determination of light transmission. As evidenced by the chart below, the system containing Klucel clearly delayed dissolution even using relatively small particles.



The system is projected to be useful in applications which require particles to withstand a combination of moisture and heat during preparation. These applications include but not limited to confections, chewing gum coatings, cereals, baked goods, pasta, snacks and dairy products in the flavor area.

In the fragrance area, the system may prove useful in delaying the solubility of fragrance particles/granules added to detergent powders and/or tablets. Delaying dissolution and fragrance release until the rinse cycle(s) may lead to an increase in fragrance deposition and substantivity.

5. Attach literature search. Indicate pertinent references below:

- US 6,187,351 but does not include hydroxypropyl cellulose as one of the polymers listed.
- A few references are indirectly related.
- Literature search is attached

6. Comments on why patent protection should be pursued:

Flavor/fragrance encapsulation in carbohydrate/gum matrices prepared via either spray-drying or melt extrusion is well known . This type of encapsulation system is sensitive to moisture and normally dissolves easily and releases flavor quickly even in cold aqueous solutions.

There is a need for flavor encapsulation systems that can withstand the high-temperature/high-moisture environments encountered in certain food-processing operations, yet still allow release of the flavor/active when the product is consumed. The matrix system described above has demonstrated these characteristics in hard, gummy and chewy candies representing a range of temperature/moisture conditions. An additional benefit is the potentially enhanced visual appeal in the finished food product since the flavor particles remain intact (as shown in the example above). The product has been demonstrated to customers such as Nabisco, Mars, and Frito and has generated significant commercial interest.

Further, the system has probable utility in fragrance encapsulation for use in products such as laundry detergent powders and laundry detergent tablets. This would be due to the slow solubility that could be expected under washing conditions, which would lead to delayed release of fragrance and ultimately improved deposition and substantivity.

While much remains to be explored with regard to the matrix system, the commercial value of the initial application – providing visible, flavored granules in hard, gummy, and chewy candies, as well as snack/gum coatings – seems clear.